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Feasibility study to manufacture briquette fuel from refinery sludge

Prepared for **Indian Oil Corporation Limited (IOCL)
Guwahati**

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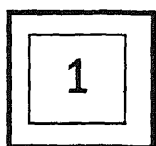
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Introduction

Petroleum refineries play a vital role in the economy development of India. Presently, there are 14 public sector refineries operating in India, with crude processing capacities ranging from 0.5 to 9.5 MTPA. Refining of the crude oil to the petroleum fuels involves multi step process consisting of storage facilities, crude desalting, distillation, catalytic cracking, hydroprocesseing, thermal processes, catalytic reforming, polymerization, alkylation, isomerization, extraction, lube oil processing, residual upgrading, blending and treating, sulphur recovery and light ends recovery. Each step of the process produces residual sludge. The maximum quantity of oily waste generated is 0.1% of the crude processed, the value ranges from 120 to 4300 MTPA for India refineries. This oily sludge is classified as hazardous waste by many countries including India. The hazardous waste can be classified into 18 categories according to hazardous waste rules, 1989. The refinery oily waste falls under hazardous waste category number 10,11,12 viz. (10) waste oil and oil emulsions, (11) tarry wastes from refining and tar residues from distillation or pyrolytic treatment, and (12) sludges arising from treatment of waste water containing heavy metals, toxic organics, oils, emulsions, spent chemicals and incineration ash.

1.1 Disposal of oil sludge

The crude oil is known to be composed of compounds made up almost entirely of carbon and hydrogen, along with some minor impurities like sulphur, nitrogen and oxygen. Due to this composition of crude oil, the residual sludge obtained during refinery process of the petroleum also contains the high calorific value hydrocarbons along with the impurities and the hazardous constituents. The physio-chemical characteristic of the oil sludge from Indian refineries is indicated in Table 1. Due to characterization of oil sludge as a hazardous waste the disposal of the oil sludge wastes in an economical and environment-friendly manner poses major challenge to all the refineries in the country. The most common practices adopted by most of the refinery within the countries are dumping the waste either in their premises or near by landfill site. However methodologies ranging from simple *insitu* treatment to biological treatment are there but the major portion of the oily waste finds its way to the

dumpsite. Such practice leads to high ecological risks as it poses potential threats to local environment like ground water contamination by heavy metals and toxic organics in soil. This practice does not allow to recover energy from the waste and at the same time the waste cannot be use as a fuel in the present form. Due to stringent environment regulations, refineries are exploring better process and methods to maximise oil recovery and better ways for disposal.

The present study sponsored by Indian Oil Corporation Limited, Guwahati is aim to develop a process for production of oil sludge mix biomass briquettes and also to quantify the environmental impact assessment of these briquettes as fuel.

Table 1. Physio-chemical characteristics of oily sludges⁽¹⁾

S.No.	Parameter	Crude tank bottom	Sludge after oil extraction	Bio-sludge	Chemical sludge
1.	Moisture at 105°C (%)	10-25	10-15	20-22	44-50
2.	PH	7-8	7-8	7-8	7-11
3.	Calorific value (Kcal/kg)	8200-9370	2700-3000	2500-3535	1600-2000
4.	Loss on ignition at 55°C (%)	95-99	40-45	85-90	33-44
5.	Oil (%)	79-90	10-15	20-25	5-7
6.	Ash (%)	1-5	61-65	15-20	67-70
7.	Metals (mg/kg)	13-225	-	26-1047	

1.2 Objectives

The broad objective of the study is to establish the process of production of biomass mix oil sludge based briquettes as fuel. This can be further subdivided into following two subheads:

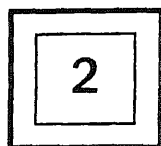
- Establishment of proper ratio of biomass and oil sludge for production of briquettes
- To quantify the different pollutants (particulate matters, sulphur dioxide, nitrogen oxides, carbon monoxide etc.) released during combustion of briquettes.
- Establish the production process for production of briquettes

1.3 Study Design

The following is the list of activities is carried out to meet the project objectives.

- 1 Data collection and literature survey
 - Visits to ministry to collect the information regarding the existing laws and regulations for the waste disposal of refineries

- Visit to Central Pollution Control Board to get information regarding
 - i Identification of different contaminants which need to be quantify for burning
 - ii Existing permissible limits for various emission stream during solid fuel burning
- 2** Procurement of raw material
 - Oil sludge from Guwahati refinery
 - Saw dust
- 3** Briquetting of oil sludge and saw dust mixture and to establish the correct mixture ratio for production of briquettes.
- 4** Experimental test runs for testing the performance of different type of briquettes which include;
 - Selection of suitable cookstove
 - Laboratory test set up for burn-cycle
 - Collection of emissions during burn cycle
- 5** Analysis of different pollutant stream
- 6** Documentation



Literature survey

In order to know the composition of the oil refinery sludge obtained from different stages of refining, their characteristics and the present methods used for its disposal, the literature survey was done in many libraries. Data was also downloaded through Internet.

The literature collected provided the characteristics of residual sludge generated in petroleum refineries. The following information was also collected:

- 1 Existing legislation and regulations, related to the waste, on the environment by Government of India
- 2 The residuals which are generated in most of the petroleum refineries
- 3 Description of residuals of concern (hazardous pollutants) present in the sludge generated at each stage of crude oil processing
- 4 Existing practices used for reduction of residuals
- 5 Various case studies of pollution prevention in petroleum refining.

Regulations by Government of India

India is the first country to provide for the protection and improvement of the environment in its constitution. The Directive Principles of State Policy, an integral element of the country's democratic principles, also contain a specific provision enunciating the State's commitment to protect the environment. Over recent years, the Government of India (GOI) has displayed a greater willingness to control industrial pollution. In 1992, the GOI issued a Policy Statement for the Abatement of Pollution, which set forth a broad outline for industrial pollution control efforts in India. The principal tenets of this policy are:

- Pollution prevention at the source.
- Encouragement, development and application of the best available practicable technical solutions.
- Recognition of the "Polluter Pays" principle.
- Focus on heavily polluted areas and river stretches.

In fulfillment of this policy, the Central Pollution Control Board (CPCB) has identified 17 industries as highly polluting, category which require priority action and petroleum refining is recognised as one of them. Pollution control for

petroleum refineries is guided by the Minimum National Standards (MINAS) that are given in the Environmental (Protection) Rules of 1986. These standards impose limits on the quality of pollutants that can be discharged for every 1000 tons of crude processed. The standards for petroleum refineries are given in Table 2.

Table 2. Emission standards from refineries ⁽²⁾

Minimal National Standards (MINAS) for oil refineries :		
Pollutant	Maximum permissible concentration (mg/l)	Maximum permissible quantum in Kilogram/1000 tons of crude processed
Oil and grease	10	7
Phenol	1	0.7
Sulphide	0.5	0.35
B.O.D.	15	10.5
Suspended solids	20	14
PH	PH shall be between 6.0 to 8.5	
Quality of Gaseous Emissions :		
Pollution parameter	Tolerance limit specified in IS 8636-1977 for emission Pollution Parameter from petroleum refineries	
Sulphur dioxide from all sources, except FCCU Regenerator	3 kg/ MT of crude, maximum	
Sulphur Dioxide from FCCU Regenerator	5.5 kg/ MT of crude, Maximum	
Particulate matters from all sources, except FCCU	125 kg/ cubic meter, Maximum	
Particulate matters from FCCU	Not specified	

The most important laws that are related to environmental protection of the petroleum refining industry include⁽³⁾:

- 1 The Water Prevention and Control of Pollution Act, 1974 and Rules 1975.
- 2 The Air Prevention and Control of Pollution Act, 1981 and Rules, 1982.
- 3 The Environment Protection Act and Rules 1986.
- 4 The Hazardous Wastes Management and Handling Rules, 1989.
- 5 Manufacture, Storage, and Import of Hazardous Chemicals Rules/Amendment Rules, 1994.
- 6 Environmental Protection Rules, 1992 - Environmental Statement.
- 7 Environmental Protection Rules. 1994 - Environmental Clearance.

Under the Environmental Protection Rules, 1994, 29 categories of projects were required to seek environmental clearance from the Central Government. Petrochemical complexes (refineries) are among the 29 categories.

2.2 Waste generation at different stages of crude refinery

Wastes from petroleum refining include free and emulsified oil from leaks, spills, tank draw-off, and other sources; waste caustic, caustic sludge and alkaline waters; acid sludge and acid waters; condensate waters from distillate separators and tank draw-off; tank bottom sludge; coke from equipment tubes, towers and other locations; acid gases; waste catalyst and filtering clays; and specialty chemicals from by-product chemicals manufacture and cooling towers.

2.3 Description of residuals and presence of hazardous pollutants⁽⁴⁾

As listed in the previous section, processing of crude in refineries is associated with release of gaseous emissions, solid waste and liquid waste. Various methodologies have been adopted to control the gaseous emissions in Indian industries.

The oil sludge, which is produced at each stage of crude processing, varies in its composition and hazardous constituents. The characteristics of few residuals and their composition are given below.

Crude oil sediment. Tank sediments are emulsions that form from solid particles, heavy oil and water that settle to the bottom of tanks over time. Hazardous constituents potentially present in crude oil tank sediments are identical to those found in crude oil. They include benzene, ethylbenzene, xylene (BTEX), sulphur, poly aromatic hydrocarbons (PAHs) and metals.

Unleaded gasoline tank sediment. This tank bottom sediments are unlike the heavy hydrocarbon sludges that typify crude oil tank sediments. Rather, small amounts of rust and scale are removed from the tank bottom. The hazardous constituents present in gasoline will also be present in the tank bottom, these include BTEX.

Residual oil tank sediment. The residual oil tank sediment consists of heavy hydrocarbons, rust and scale from process pipes and reactor and entrapped oil that settles to the bottom of the tank. Hazardous constituents potentially present in residual oil tank sludge are similar to those found in crude oil tank sludge. They include BTEX, sulphur, PAHs and metals.

Clarified slurry oil (CSO) sediments or filter solids. This is generated from the intermittent cleaning of CSO storage tanks. Hazardous constituents potentially present in CSO tank sludge are similar to those found in crude oil tank sediments. They include BTEX, sulphur, PAHs and metals. In addition, Fines from catalytic cracking (FCC) catalyst will be present in the CSO. This solid will settle out in the tanks and add significantly to the sludge volume.

Desalting sludge. Hazardous constituents potentially presenting in desalting sludge are benzene and PAHs. Hydrotreating/ hydro-refining/ hydrocracking catalyst: It is mainly to remove sulphur, nitrogen and metals and to saturate olefins and aromatics. Hazardous constituents include the base materials like cobalt, nickel, molybdenum, and aluminium. Other than this it also contains hydrogen sulphide, and other metals or organic present in the hydrocarbon feed.

2.4 *Methods used for reduction of pollution*⁽⁵⁾

It is now mandatory to adopt some technique for pollution and prevention and reduction. The petroleum industry has identified a number of options that refineries could undertake to reduce pollution.

2.4.1 Pollution prevention options

Pollution prevention options are usually categorised into four areas: good house keeping practices, changes in production processes, input substitution, and product reformulation. The options described here include only the first three of these categories.

Good house keeping practices. Good house keeping practices prevent waste by better handling of both inputs and wastes without making significant modifications to current production technology. If the inputs are handled better, they are less likely to become wastes and inadvertently through spills or outdating. If wastes are handled better, they can be managed in the most cost-effective manner. Some good housekeeping options that have been identified in the petroleum industry include:

- Segregate process waste streams
- Control solids entering sewers
- Identify benzene sources and install upstream water treatment
- Train personnel to reduce solids in sewers
- Train personnel to prevent soil contamination

Production process modifications. Production process modifications involve capital equipment, layout and process changes that reduce the amount or toxicity of wastes that are generated. Some options for process modifications in the petroleum refining industry include:

- Install vapour recovery for barge loading
- Replace old boilers
- Reduce the use of 55 gallon drums
- Install high pressure power washer

- Refurbish or eliminate underground piping
- Eliminate use of open ponds
- Remove unnecessary storage tanks from service
- Place secondary seals on storage tanks
- Establish leak detection and repair program
- Minimize solids leaving the desalter
- Minimize cooling tower blowdown
- Control of heat exchanger cleaning solids
- Control of surfactants in waste water
- Install rupture discs and plugs

Material substitution options

- Use of non-hazardous degreasers
- Eliminate chromates as an anti-corrosive
- Use of high quality catalysts
- Replace ceramic catalyst support with activated alumina supports

2.4.2 Recycling options

Although pollution is reduced more if wastes are prevented in the first place, a next best option for reducing pollution is to treat wastes so that they can be transformed into useful products. Some recycling options identified for the petroleum industry include:

- Recycle and regenerate spent caustics: caustics used to absorb and remove hydrogen sulphide and phenol contaminants from intermediate and final product streams can be recycled.
- Use of oily sludges as feedstock: many oil sludges can be sent to a coking unit or the crude distillation unit where it becomes part of the refinery products.
- Control and reuse fluidized bed catalytic cracking unit (FCCU) and coke fines: coke fines can also be recycled for fuel use.
- Recycle lab samples: lab samples can be recycled to the oil recovery system

2.4.3 Treatment options

When the pollution prevention and recycling options are not economically viable, pollution can still be reduced by treating wastes so that they are transformed in to less environmentally harmful wastes or can be disposed of in a less environmentally harmful media. Some treatment options identified for the petroleum industry include:

- Thermal treatment of applicable sludges

- Improve recovery of oils from oily sludges
- Reduce the generation of tank bottoms
- Regenerate or eliminate filtration clay
- Minimize FCCU decant oil sludge

The main objective of the current study was to establish the process of production of oil sludge briquettes and to study its performance characteristic as fuel. In order to complete the project objectives in the stipulated time frame, the project team members were regularly holding the project meeting to review the progress made and to discuss the future action plan. In this chapter, methodology and protocols used for assessing the various combustion characteristic of the fuel are discussed.

3.1 Proximate analysis (moisture content and ash content)

Moisture content and ash content of the oil sludge and various briquettes formed was determined according to ASTM standards D3172-3173. Moisture content was determined by drying the weighed amount of sample in open petri-plate kept at 105°C in an oven till the consistency of weight is obtained. The difference between the initial and final weights is taken as moisture content of the fuel. The sample is then transferred in to an open crucible and gradually heated to 750°C in a Muffle furnace and is maintained for four to five hours. The weight of the residue represents the ash content of the sample.

3.2 Ultimate analysis

The ultimate analysis of a fuel is important parameter to assess the fuel value by measuring carbon, hydrogen, nitrogen and sulphur content. The ultimate analysis of oil sludge, oil sludge-biomass briquettes, oil sludge-petroleum coke dust briquettes was carried out through M/s Shri Ram Institute of Industrial Research, Delhi. The carbon and hydrogen content is determined using CHN analyser, nitrogen by kjeldhal apparatus, sulphur by gravimetric method and oxygen is calculated by difference basis.

3.3 Calorific value

Calorific value of oven dried samples was estimated in a standard bomb calorimeter. Benzoic acid was used for standardisation of the calorimeter. One gram of sample was taken in a crucible and made into a pellet and the initial weight was noted. The pellet was placed in the bomb, which was then pressurised up to 18

atmosphere by oxygen. The bomb was then placed in a vessel containing a measured quantity of water. The ignition circuit was connected and the initial water temperature was noted. After ignition the temperature of the water is noted every minute interval till a constant temperature was recorded. The pressure was released and the length of unburnt fuse wire was measured. The calorific value was calculated using following formulae:

$$(t_c \times w) - (m + n) / \text{weight of sample} = \text{kJ/kg}$$

where,

t_c = temperature rise (C)

w = apparent heat capacity by benzoic acid (J)

m = calorific value of thread (J)

n = Calorific value of Nichrome ignition wire (J)

The apparent heat capacity by benzoic acid (w), calorific value of the thread (m) and nichrome wire (n) was provided by the instrument supplier.

3.4 Calorific value of producer gas

Calorific value of producer gas was measured using an INSREF Junker's Gas Calorimeter, which can be used to analyse the gases with calorific range of 1000-26000 kcal/m³. The method is based on measurement of heat gained, in the form of temperature rise, by the known amount of water due to combustion of known amount of sampling gas.

The formula used to calculate the calorific value to the test gas is

$$C.V. = \frac{V_w}{V_g} \times (t_2 - t_1) \times 100$$

where,

C.V. is Calorific Value of gas in Kilocalories/cubic metre;

V_g is volume of gas in litres consumed during test period;

V_w is volume of water in litres passed during test period;

t_2 is outlet water temperature in °C; and

t_1 is inlet water temperature in °C.

3.5 Flue gas sampling

The sampling for total suspended particulate matter (SPM) needs to be carried out in isokinetic mode. Therefore, initially velocity of the flue gas was measured at upper end of the chimney with an inclined vertical manometer fitted at the panel of the stack monitoring kit. Pitot tube, which is attached to the manometer for velocity measurement, was placed in the chimney through the hole at the upper end and its position was adjusted to get the maximum deflection in the manometer. Maximum deflection was found at an axis along the direction of airflow in the chimney. The deflection in the manometer fluid is used to estimate the velocity of the flue gas in chimney. Velocity of the flue gas in present chimney of 70 cm height and 13.2 cm diameter was found to be 2.2 m/s.

The measurement of velocity along with selection of probe for sampling led to isokinetic sampling rate determination. In present study sampling was done with a probe of 1/2" I.D and at the rate of 9.4 lpm, for total duration of 90 min. The gas was sampled from the same point in the chimney where velocity measurement was carried out. The Envirotech stack sampler APM 620 with pre-weighed glass microfibre thimble and two impinger tubes with absorbing medium for SO_x were used for sampling of SPM and SO_x respectively. The gas was also filled in the tedlar bag for determination of CO measurement using gas chromatograph. Method followed for determination of these parameters given in Annexure-I.

3.6 Particulate measurement

As described earlier, approximately 1-2 m³ of flue gas sampled is passed at pre-

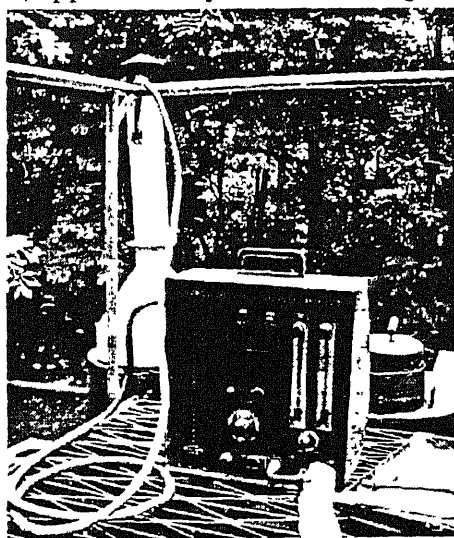


Figure 1 Particulate measurement in chimney using stack monitoring kit

determined isokinetic sampling rate through a pre-weighed micro glass fibre thimble, placed in the stack monitoring kit. The flow rate was monitored both at the start and end of the sampling. The difference in the initial and final weight gives the mass of dust collected in the thimble, which represents the amount of suspended particulate matter (SPM) in the volume of gas sampled. The method of conditioning and weighing of filter is described in annexure-I

3.7 GC analysis

Carbon monoxide and particulate in the flue gas have potential health hazards such as conjunctivitis, acute respiratory infections and upper respirator irritation. Carbon monoxide has indirect greenhouse effect by coupling with atmospheric methane. Moreover it also affects the concentration of hydroxyl (OH) radical and cycle of tropospheric ozone. In order to measure the concentration of CO in flue gas samples a gas chromatograph (GC) setup was used. A system of GC- flame ionization detector (FID)-Methanizer has been used for analysis of CO. For separation of the CO, a carbosphere packed column has been used in the FID. The separated CO was converted into methane by hydrogen at 375°C in a nickel catalytic device called methanizer to obtain methane, which can then be determined by FID. The GC was periodically calibrated using Spancan standard calibration mixture.

3.8 Nitrogen oxide measurement

The sample of flue gas is collected in a bladder and transferred into an evacuated flask, containing dilute sulfuric acid-hydrogen peroxide absorbing solution. The nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenol disulphonic acid (PDS) procedure. Detailed procedure along with chemical preparation and spectrometer calibration is given in annexure-I.

3.9 Sulphur oxide measurement

The sulphur oxide in the flue gas was measured based on hydrogen peroxide method. It is based on the principle that when the SO₂ containing flue gases are bubbled into the hydrogen peroxide, it is rapidly oxidized into non-volatile H₂SO₄, which can be quantified by simple acid-base titration.

In order to measure the concentration of SO₂ in the flue gas, the stack monitoring kit was used. Two impinger tubes containing SO₂ absorbing solution are kept in series to the thimble holder. During sampling CO₂ present in the flue gas may also get absorbed and form carbonic acid. In order to overcome this interference, pH of the absorbing reagent was adjusted to 5. The flue gas after

passing through the filter in the thimble holder bubbles through this absorbing solution. The concentration of sulphur oxide was measured by acid base titration.

3.10 Poly aromatic hydrocarbons

After gravimetric quantification of suspended particulate matter, the thimble was cut in two halves vertically. The aromatic fraction was extracted from suspended particulate using one half in benzene. The extracted sample was again dissolved in toluene and filtered. The aliquot is analysed by gas chromatography with flame ionization detector (GC-FID; 5890 series II; Hewlett Packard) using a 30 m long wide bore DB5.625 column (0.25 mm i.d, 0.25 μ m film thickness). During analysis the injector and detector temperature of GC were maintained at 300°C and the oven temperature was program to rise from 80 to 240°C at the rate of 5°C/min and maintains at 240°C for 30 minutes. Area under the peak in comparison to known concentration of standard represents the concentration of individual component.

Briquetting is a technology which compresses loose biomass into densified form called "briquettes". The technology of briquetting can broadly be divided into two types namely low-density and high-density processes. The former technology makes use of a binder to keep the material together, while in the latter method the high pressure is generated in the process which causes the material to bind. In India, the briquetting industry was started in 1981 with introduction of both low and high-density technology.

Low-pressure briquetting is generally been carried out for high moisture content materials and with the use of binder. The low-pressure briquetting technology is an indigenous developed technology and mainly used for pyrolysis biomass (char) or coal dust with binder. It is possible to briquettes any material of biomass origin and considerable experience has now been build up within India with a wide range of residues such as

- Field residues e.g. cotton stalk, wheat straw
- Processing residues e.g. ricehusk, coffee husk, baggasse
- Forestry and wood processing waste e.g. saw dust
- Domestic and industrial wastes e.g. tobacco waste, herbal waste
- Coal dust

Residues normally vary in their physical and chemical properties and therefore require different densification processes. In addition for some of the residues the pretreatment is required before briquetting such as drying, purification, size reduction, to ensure the production of a good quality briquette. The most important parameters influencing the briquette quality are:

- Moisture content
- Particle size
- Shape

Moisture content plays an important role in binding the particles together and too high moisture content causes steam to form during densification process which will then weaken the structure of the briquette. Each briquetting technology has its own specific requirements for moisture content but range lies within 5 to 20%.

In this Chapter, the process of production of oil sludge mix with biomass briquettes production is described in detail.

4.1 *Procurement of Raw material*

The project was initiated with the procurement of main raw material (oil sludge, from IOCL Guwahati. TERI had arranged the procurement of oil sludge through their North-East centre situated at Guwahati. The oil sludge of about 2000 kg filled in ten plastic barrels (nine barrels from dump yard and one barrel fresh sludge) along with a bag of petroleum coke dust was transported to Delhi. Since the movement of vehicle is restricted in the IOCL campus, the material was moved with a handcart inside the campus and then sent to TERI Gual Pahari Campus through road transport.

The sludge obtained from the refinery is in thick viscous slurry form and black in colour. The photograph of the plastic containers containing the sludge is shown in Figure-2.

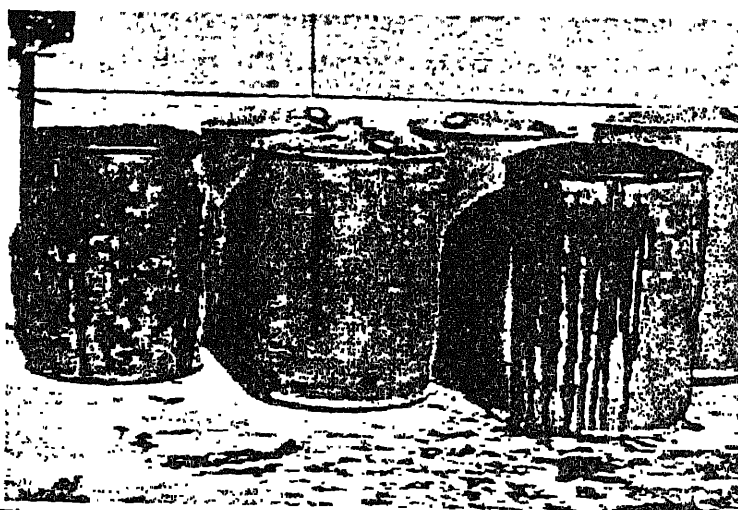


Figure 2. Oil sludge obtained from IOCL Guwahati

The oil sludge obtained from the IOCL, Guwahati refinery was found to be having more than 30% liquid content (water and oil together) and observed to be not suitable for briquetting in the present form. Therefore, it was proposed to establish the process of production of oil sludge mix with biomass briquettes using briquetting technology. As the sludge is having binding quality, therefore, a low-pressure briquetting press was found to be the most suitable option for this purpose. Hence, a screw briquetting press installed at TERI Gual Pahari campus was selected for experimentation.

4.2 *Preparation of briquettes*

The work on production of oil sludge mix with sawdust briquettes was started at TERI Gual Pahari campus. The mixture of oil sludge and biomass were

briquetted using screw briquetting press. The photograph of the briquetting machine used is shown in Figure 3. Different combination ratio of oil sludge and biomass mixture were tried for production of briquettes. Finally, mixture combination of 1:3 and 1:2 (saw dust: oil sludge) ratio were found to be the most successful both in terms of production and quality. The green briquettes produced using screw press are then placed in open for sun drying. After about two days of sun drying the briquettes were found suitable for subsequent use. The photograph of the biomass mix oil sludge briquettes is shown in Figure 4.

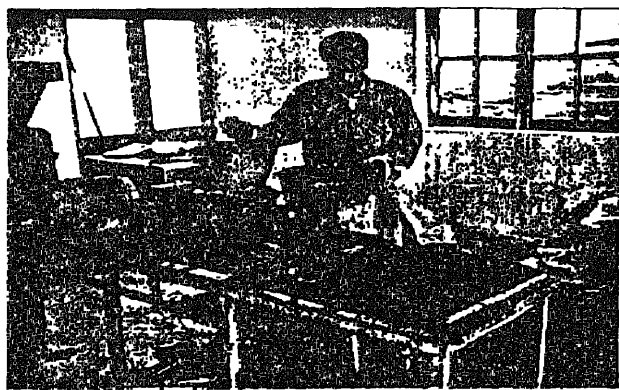


Figure 3 Production of oil sludge-biomass mix briquettes at Gual Pahari



Figure 4 Sun drying of green briquettes

4.3 Visit of IOCL officials to Gual Pahari

A team led by Mr Prithwiraj Sur, DGM(T), Mr P.K. Goswami, Sr Manager (Energy and safety audit) and Mr Amalendu Kumar, Chief Instrumentation Manager from IOCL Guwahati visited the TERI on 11 and 12 January 2001 to review the progress of the project and to discuss the future course of action. The team also made a visit to TERI's Gual Pahari campus to see the production of oil sludge briquettes. The team had also seen a demonstration of using these briquettes in a down draft gasifier. During the visit, the team pointed out that it would be difficult for them to procure the sawdust for production of these briquettes on commercial scale operation. They further informed that the petroleum coke is generated as waste during de-coking process of crude oil refining and also available in huge quantity at IOCL, Guwahati refinery. They suggested the TERI team to look into the feasibility of using petroleum coke dust in the production of briquettes in place of saw dust. Based on the field visit the following points were agreed upon:

- To study the feasibility of using petroleum coke dust in place of biomass for production of the briquettes

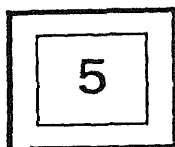
- The study will focus on assessment of emission characteristic of these briquettes in gasification mode.
- The study will quantify the emissions such as SO_x, NO_x, and PAHs (Poly Aromatic Carbons) in flue gases and heavy metals like Ni, Vanadium and lead in ash.

The minutes of the meeting is given in Annexure-II

4.4 *Oil sludge petroleum coke dust briquettes production*

An additional quantity of about 200 kg of petroleum coke dust was arranged from Guwahati refinery for the purpose of production of oil sludge mix petroleum coke dust briquettes. Similar to the oil sludge-biomass mix briquettes, the trial runs have been carried out with different composition of oil sludge mix petroleum coke dust. Since the particle size of the petroleum coke dust is very fine, therefore it was found to be more difficult to form the oil sludge-petroleum coke dust briquettes. These briquettes tend to form surface cracks during drying stage probably due to release of trap moisture. To overcome this problem, the composition of coke dust was increased gradually to achieve a suitable mixing ratio of petroleum coke dust and oil sludge mixture. Subsequently, a successful composition of 3:2 (coke dust and oil sludge) was found to be the most suitable. However, during drying the briquettes still tend to form few minor surface cracks.

The detailed chemical analysis and combustion characteristic of both types of briquettes were carried out. The combustion performance of these briquettes as fuel is discussed in detailed in the following section of the report.



Environmental impact assessment as fuel

As discussed in the earlier chapter the stable briquettes has been produced with both oil sludge mix with biomass or petroleum coke dust. Since the oil sludge categories as the hazardous waste as per the pollution control norms, it is primitive to study their combustion performance as fuel and to quantify the different combustible stream of these briquettes as fuel. In this chapter, the combustion performance of these briquettes as fuel both in cookstove and gasifier is discussed in detail.

5.1 Pilot testing of briquettes in stove

It was decided to test the performance of these briquettes in cookstove to measure different pollutants. For this purpose, a trial test set-up was created to experiment the suitability of these briquettes as fuel with the following main objectives.

- To observe the combustion performance
- To finalize the experiment protocols for measuring the various emissions.

A new improved cookstove was designed and has been fabricated mainly to use briquette fuel. The outside body of the stove is made from MS inside lined with castable insulation with provisions for supply of secondary air required during the burning process. A MS grate of 100 mm diameter is placed at the bottom having manual shaking arrangement. The primary air for burning the fuel is supplied from the bottom of the grate. The combustible gases generated during the combustion process, and secondary air from the insulation holes, mix together at the mixing chamber and burn above the fuel bed. A fuel charging port is placed at one side for feeding the fuel even during running. A 4" diameter air port is also placed mainly to supply forced air from a centrifugal blower if required.

A photograph of the cookstove is shown in Figure 5. The trial runs on oil sludge briquettes were carried out using the cookstove.



Figure 5. Stove designed & used for the combustion of oil sludge mix briquettes

5.1.1 Experimental set-up

A known quantity of oil sludge - biomass briquettes is loaded into the cookstove. The cookstove was placed at the centre of the hood in order to ensure that the combustible gases will pass through the hood. An aluminium pan of 32 cm diameter and 17 cm height is filled with 5 litre of water. A mercury jeal thermometer with 0.5°C accuracy is placed in the pan through a hole in the lid using rubber cork for measuring the water temperature. The cookstove is run on natural draft mode and fuel is ignited from the bottom of the grate using gas lighter. 10 ml of kerosene was kept aside for ignition if required. The temperature of water is monitored every minute and noted in the data sheet (see Annexure-III) till the water is boiled. Once the water starts boiling, the lid of the pan is removed for evaporation. Wait till all the fuel got exhausted and then both ash and remaining water were weighed. During these experiments the combustion performance and burning rate of the fuel was monitored.

5.1.2 Observations

A total of three experiments were conducted on natural draft mode to study the performance of briquettes. The briquettes can be ignited with relative ease, but during trial runs it was observed that emissions from briquettes were higher, especially during initial stages. The fuel burns with constant flame without any tending. No deformation of the briquettes was observed and complete ash powder with no traces of carbon was found at the end of combustion. Huge amount of thick smoke with sooty flame was observed. The high amount of heat



Figure 6. Burning of oil sludge
biomass briquettes in cook stove

release rate per unit area of grate is probably due to higher calorific value of the briquettes resulting in insufficient air supply during combustion. This is confirmed by high amount of particulate matter (unburnt particles) which can be seen in the smoke and latter settled down on the floor. A low burning rate of 0.75 kg/h of briquettes was observed.

The air supply in natural draft mode is limited therefore, it was decided to test the combustion performance of these briquettes in the forced air mode with the use of a centrifugal blower.

5.1.3 Combustion experiments using a blower

Due to limitation of increased supply of air for combustion in natural draft mode, centrifugal blower having 0.25 hp and 0.5 hp capacity was used for combustion. The combustion performance tests were carried out using the blowers independently. The noticeable difference was observed in reduction of smoke and soot level in the flue gases compared with earlier natural mode operation. At the same time burning rate has been increased to 1.75 and 2.2 kg/h with 0.25 hp and 0.5 hp blower respectively. However useful power of the stove is reduced considerably. This is mainly due to high flue gases losses resulted due to several fold increase in flue temperature. It is thus apparent that the combustion characteristics of these briquettes have to be studied vigorously in order to design a suitable combustion equipment for these briquettes.

The main difference between these briquettes and other fuel is in the proximate analysis. The oil sludge briquettes have higher amounts of oil content, lower ash content and lower amount of sulphur. Therefore, it was decided to test the performance of these briquettes in gasification mode which also allows briquettes to compete with furnace oil, LDO etc. For this purpose, briquettes were tested in a down draft, throatless gasifier developed by TERI. This mode of utilization of briquettes will be discussed in detail in the following sections.

5.2 Trial testing of briquettes in gasifier

The gasifier is a device, which converts solid fuel into a combustible gas called producer gas. The process of gasification is carried out at sub-stoichiometric conditions with air-to-fuel ratio of being 1.5:1. In the gasifier, solid fuel pieces undergo drying, pyrolysis, combustion and reduction process and yield a combustible gas having a calorific value of 1000-1200 kcal/Nm³ with an average composition of:

CO	:	18-20%
H ₂	:	18-22%
CH ₄	:	1-4%
CO ₂	:	11-13%
N ₂	:	45-55%

The producer gas can be used for both thermal and power generation applications. Flame temperature as high as 1200°C can be obtained by optimal pre-mixing of air and gas. The producer gas can be used to operate a diesel engine on dual fuel mode and gas engine on 100% producer gas. The gas can replace 80 to 85% diesel consumption at rated loads on dual fuel mode. BETA group of TERI, has been doing extensive work in the development of gasifier technology for efficient utilization of biomass, agricultural residues, industrial waste etc. The group is actively involved in the development of biomass gasifier systems for thermal applications in small-and medium-scale industries. Ovens kilns, and furnaces based on biomass gasifier technologies have been developed for industries such as silk reeling, textile dyeing, magnesium chloride production, brick drying and spice drying. These systems have demonstrated fuel savings of the order of 45-60%, along with significant increase in overall productivity at the enterprise level. The group has successfully installed more than 100 system having capacity ranging from 25,000 kcal/h to 4,00,000 kcal/h with a total cumulative installed capacity of more than 5 MW.

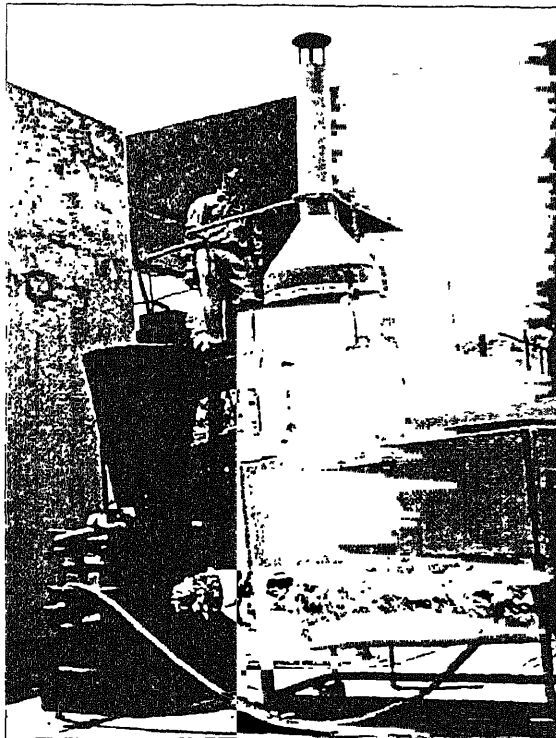


Figure 7. Gasifier system used for testing of oil sludge briquettes

For the purpose of testing the performance of oil sludge-biomass briquettes, a down draft, throatless gasifier having capacity 25,000 kcal/h coupled with silk reeling cooking oven installed at Gual Pahari was used (Figure 7). The briquettes filled into the gasifier from the fuel-charging door and fuel is ignited from the ignition ports by placing the flame torch. Initially the gas is flared from the flaring port till the combustible gas is obtained. The gas is then diverted into the burner by closing the flaring port. No smoke and soot was observed during the burning of gas.

The flame temperature observed was comparatively higher probably due to high calorific value of the briquettes. No operational difficulties were observed during the trial test runs. Further it was decided to carry out the emission study of these briquettes in gasifier mode only.

5.3 Emission performance study

As discussed earlier, the performance of these briquettes was carried out in a down draft gasifier having 25,000 kcal/h capacity. The combustion characteristics of both the types of briquettes (oil sludge-biomass and oil sludge-petroleum coke dust) were tested.

5.3.1 Proximate analysis

The proximate analysis of the raw material and fuel briquettes are carried out at biochemical laboratory of TERI at Gual Pahari. While the ultimate analysis was carried out from the M/s Shri Ram Institute of Industrial Research, Delhi a reputed organized laboratory. The result of proximate analysis of oil sludge, biomass mix and petroleum coke dust mix briquettes are summarized in Table 3. It can be seen from the table that ash content of oil sludge–petroleum coke dust briquettes is comparatively lower than with oil sludge–biomass briquettes which may be due to low ash content of petroleum pet coke of the order of 0.5%.

Table 3. Proximate analysis of the oil sludge briquettes

Description	Raw sludge	Oil sludge-saw dust briquette	Oil sludge-Petroleum coke dust briquette	Petroleum coke dust
Ash content (% of TS)	3.4	6.9-9.4	2.1-2.6	0.47-0.76
Moisture (%)	61.2-67.1	20.6	21.3	5.4

5.3.2 Ultimate analysis of briquettes

The ultimate analysis of oil sludge, oil sludge–biomass and oil sludge–petroleum coke dust has been carried out from M/s Shri Ram Institute of Industrial Research, Delhi. The parameters such as C₂, H₂, N₂, O and S were analyzed and given in Table 4. It can be seen from the table that the percentage of fixed Carbon, Hydrogen is substantially higher in case of oil sludge–petroleum coke dust briquettes which results in higher calorific value of the fuel. However, the sulphur content is of the order of 0.5% in all the fuels. The reports of the test result is given in Annexure III.

Table 4. The fuel analysis results

Description	Raw sludge	Oil sludge-saw dust briquette	Oil sludge- Coke dust briquette	Ash left after Oil sludge-Coke dust briquette combustion
Ash content (%)	3.4	8.5	2.3	----
Carbon (%)	60	72.5	85.3	67.2
Hydrogen (%)	9	1.4	10.6	7
Nitrogen (%)	3.9	0.9	1.3	2.5
Sulphur (%)	0.5	0.4	0.5	0.3
Oxygen (%)	23.2	Nil	Nil	14.3

Calorific value	4340	5325	6321	-----
(kcal/kg)				

5.3.3 Gasification of the briquettes

Both oil sludge–biomass and oil sludge–petroleum coke dust briquettes were successfully trial tested in the gasifier. The calorific value of the producer gas obtained with oil sludge–petroleum coke dust briquettes is about 1199 kcal/Nm³. The composition analysis of producer gas is carried out using gas chromatograph. For this purpose a sampling port was made, before the burners, in the producer gas carrying duct. The producer gas is sucked from the sampling port in a pre-vacuumed glass tube. The glass tube has a port with rubber septum from where sample can be withdrawn using a gas tight syringe. This grab sample of producer gas in the tube is used for multiple injections in the gas chromatograph for composition analysis. The results of composition of constituents are:

CO	:	12.4%
H ₂	:	20.4%
CO ₂	:	2.4%
CH ₄	:	1.8%
N ₂	:	61.9%

5.3.4 Emission assessment

The parameters studied for emission assessments during combustion of briquettes in gasifier were suspended particulate matter, Carbon monoxide, NO_x, SO_x, poly aromatic hydrocarbons and heavy metals like lead, cadmium, nickel, and chromium. All the measurements were carried out by taking flue gas sample from the chimney. Temperature and velocity measurements were carried out during each run, before starting the emission measurements, using Envirotech velocity monitor. Isokinetic sampling was carried out for SPM using Envirotech stack sampler APM 620. Preconditioned and weighed glass fibre thimbles were used for particle collection. The SPM levels were calculated by gravimetric method. PAH and heavy metals analysis were carried out using gas chromatograph with capillary column and atomic adsorption spectrophotometer with flame (Analytic Jee) respectively. SO₂ and NO₂ measurements were carried out according to hydrogen peroxide and EPA method 7-1 respectively. The measurement of CO in the flue gas was measured using gas chromatograph.

5.4 Results and discussion

The concentrations of different pollutant emissions during oil sludge-biomass mix and oil sludge-petroleum coke dust mix briquettes combustion in the down draft gasification process are summarised in table 5. The combustion and emission study of these briquettes is being carried out for the first time in India. Non availability of standard values under any environment protection rules for pollutant emissions during combustion of oil sludge is a hurdle to meet the objective. Therefore a meeting was organised with CPCB officials to discuss the results of the study and to consider their view point. CPCB officials suggested to compare the emission values to the standard available for boilers. Since the boiler standards are only for SPM other values are compared with prescribed EPA standards for combustion of oil sludge in incinerator.

Table 5. Emissions during combustion in gasifier

Description	Oil Sludge- Biomass briquette	Oil sludge-coal dust briquette
Carbon monoxide (ppm)	669	242
Methane (ppm)	115	No trace
Suspended particulate matter (mg/Nm ³)	109	71
SOx (mg/Nm ³)	192	1047
NOx (mg/Nm ³)	8 (7-9)	21 (20-21)
PAH	BDL	BDL
Chromium (ppm)	0.35 (0.23-0.462)	0.23 (0.19 - 0.26)
Nickel (ppm)	0.09 (BDL - 0.163)	0.06 (BDL - 0.12)
Lead (ppm)	0.24 (0.15 - 0.98)	0.73 (0.50 - 1.07)
Cadmium (ppm)	BDL	BDL

The concentration of carbon monoxide emission was found to be lesser for petroleum coke dust based briquettes combustion compared to biomass based briquettes. But the CO emission levels are higher for both types of briquettes when compared to the standard emission level of 199.2 ppm, specified by emission standard for the coal fired boiler of 0.5 to 1 MW plant by European Union data base 1999. The emission of methane from petroleum coke dust based oil sludge briquettes is under the prescribed limits against the value of 101.7 ppm by the European union database. The levels of poly aromatic hydrocarbon emission in the particulate were found to be below detectable limits of the gas chromatograph. As there may be a little possibility of their presence in the gaseous form further refinement in the sampling procedure is needed. The high

emission of product of incomplete combustion (PIC) such as CO and CH₄ from biomass briquettes may be due inadequate oxygen supply. The level of suspended particulate matter emission during combustion of briquettes is much lower than the specified level of 1600 mg/Nm³ under the Environment protection rules, 1986 for small boilers of capacity of less than 2 ton/h. The SO₂ emission level for petroleum coke dust-oil sludge briquettes is very high but this high concentration can be taken care by installing the appropriate stack height which can be calculated using the below mentioned formulae given by Environment (protection) rules, 1986:

$$H \text{ (stack height)} = 14 (Q)^{0.3}$$

where,

Q is emission rate of SO₂ in kg/h

H is stack height in meters.

In the present study, the briquettes were tested in a gasifier system where the velocity is 2.2 m/s and the chimney dia is 13 cm. The required minimum stack height is estimated to be about 4 meters for oil sludge-biomass briquettes and 9 meters in case of oil sludge-petroleum coke dust briquettes.

The present study has successfully demonstrated the use of oil sludge for the production of oil sludge–biomass (3:1) briquettes at the laboratory scale. On the extended scope of the project, the production of oil sludge – petroleum coke dust briquettes has also been achieved successfully. The process of production of briquettes involves uniform mixing of raw material in the correct proportion, briquetting of mixture, sun drying of briquettes and storing of briquettes. On the commercial scale, the process of production of oil sludge – petroleum coke dust mixture is also tried successfully with the help of M/s Hi-Tech Agro Pvt. Ltd. Based on the outcome of the project, the study recommend to install a 2-tonne/h output capacity commercial scale palletizing plant at IOCL, Guwahati.

The process of palletizing is the physical transformation of the loose material into a compactly compressed form. The compact form will result into higher density of material which increases its combustion efficiency. The resultant pallet fuel has high calorific value of more than 6,000 kcal/kg and low ash content of less than 1%. Hence, these pellets fuel is an ideal and ready substitute for coal, firewood, lignite etc. and can easily replaces these conventional fuels for heat, steam generation etc.. The pellets can also be utilize in the gasification process and replace fossils fuels like diesel, F.O, kerosene etc.. The other advantages of these pallets will be ease of handling and storage and can be packed in bags there by reducing labour costs in handling, sorting and breaking.

The concept of pelletising is fairly old. Coal dust and other materials have been palletised for a number of years using binder. The plant and machinery required are dependent on the feedstock being processed. A typical palletizing plant would consists of pelletising machine, intermediate storage bin, material handling equipment, hot air generator and dryer.

Layout of the proposed commercial plant is given in Figure 8.

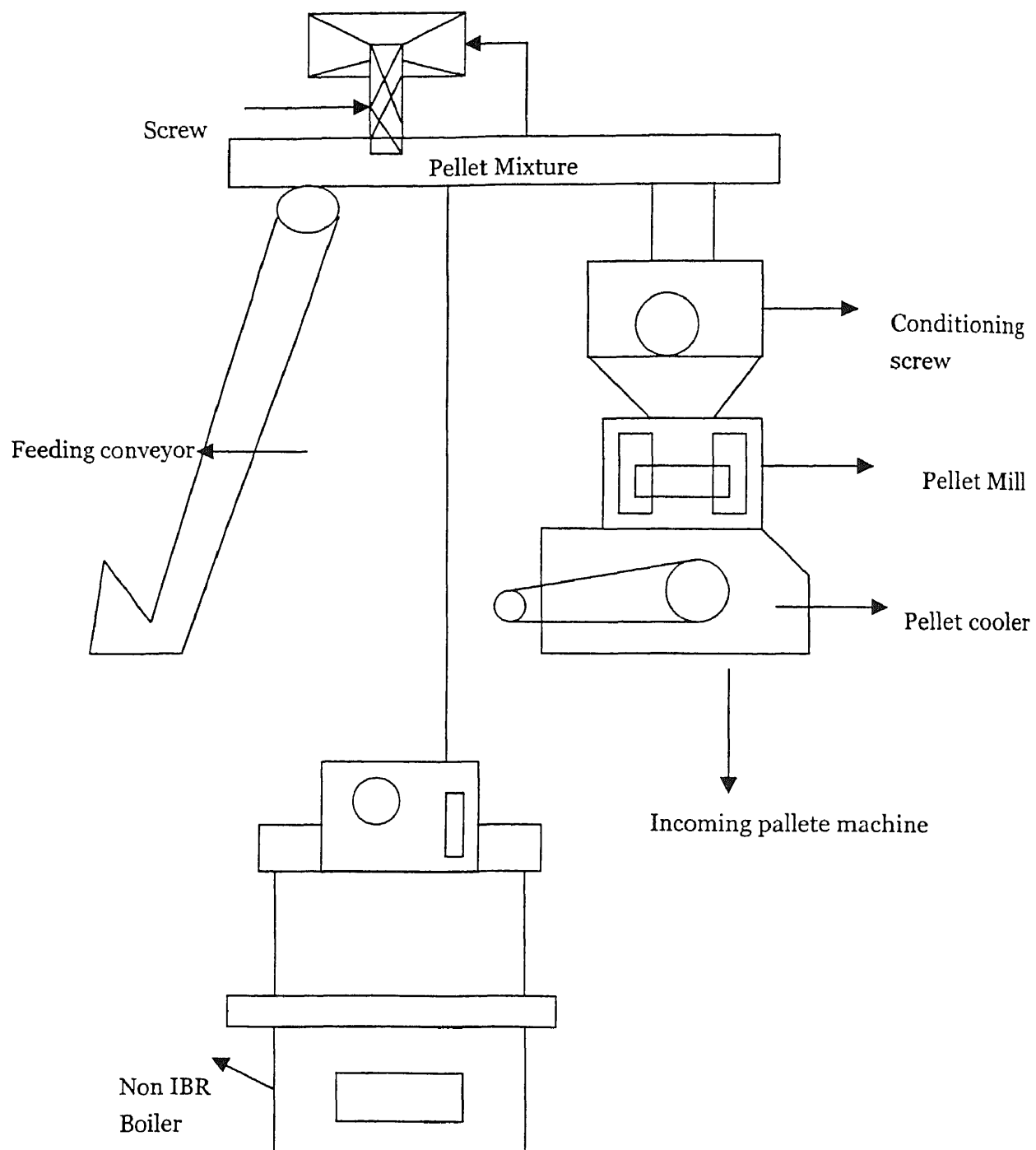


Figure 8: Layout of the proposed commercial plant

For this purpose, a detailed investment proposal will be submitted to IOCL for consideration separately. Since the oil sludge is a thick viscous sticky material the challenge will be in designing a suitable mixing device for this application. However, following points still have to be discussed in detail before finalizing the plant and machinery configuration.

- The level of automation needed for plant and its cost consideration.
- Whether such a plant will be operated by IOCL staff or by third party.
- Total plant layout along with material flow and storage of different raw materials and briquettes within the plant.

It seems that these briquettes can be used for substitution of high grade coal used in many industrial applications such as mini-cement plants, lime kilns, foundry, potteries and industrial boilers. Therefore, it is proposed to carryout a detailed market assessment study of these briquettes as fuel for various potential users. The study would help in establishing the cost & price of the briquettes, potential clusters, packing and forwarding requirement of the fuel etc. In addition the socio-economic factors of using these briquettes as fuel can also be studied.

In order to make this technology viable and proper use of these briquettes the discussion was held with the Central Pollution Control Board officials who are dealing with the hazardous waste management. They indicate to the team that coal dust based briquettes are more convenient to use as a fuel due to less emission of carbon monoxide, methane and suspended particulate matter. Although the SO₂ emissions are higher but the high concentration can be managed by installing a proper height of stack. The use of these briquettes at the household cooking activities is not suggested due to unregulated and high emissions level.

Further to this emission study, the officials of pollution board suggested following more parameters can be studied.

- Chlorine gas : The presence of chlorine gas or hydrogen chloride indicates the combustion of chlorinated organics. This gas has health risk and is a large component in the formation of acid rain. Moreover the presence of chlorine gas in stack also shows the possibility of presence of chlorinated aromatic hydrocarbons in the stack which have high health impacts. Thus it is necessary to keep a check on the chlorine gas and related compounds in case chlorine is found in the stack.

- Organic content: Since the combustion of hazardous waste should destroy the organic content it is necessary to verify the efficiency of combustion by measuring organic content in the raw material and in the emissions.
- Inorganic content: It was suggested to carry out the mass spectrophotometry of the raw material to know the total inorganic components. This will ultimately help to concentrate the emission study for particular toxic metals.

Therefore the present study recommends to analyse these parameters in the extended testing.

Policy recommendations

- The industry, which is in the process of producing fuel using hazardous waste material, should need to be get clearance from state pollution boards.
- These briquettes are not suitable for the household cooking appliances
- The briquette fuel could be used by industries with a minimum required stack height
- The emission levels should be monitored time to time for safety, health and environment impact consideration

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Annexure-I**A. Proximate analysis (moisture content and ash content)****Procedure**

1. Start the oven after setting the temperature to 105°C
2. Silica crucibles are cleaned properly
3. Around 1.5 g of sample is added to the crucible using clean spatula and combined weight of crucible and sample is taken in 1 mg accuracy.
4. Similarly sample is taken in three crucibles.
5. Samples are kept in oven, the temperature of which is already maintained at 105 °C.
6. Weight of samples is regularly monitored after an interval of 3-4 hours till it attains constant weight.
7. Note the final weight of the samples.
8. The sample is then gradually heated to 750 °C in a muffle furnace
9. Final weight is recorded after cooling of sample
10. The weight of the residue represented the ash content of the sample.

Calculation

$$\% \text{ moisture content (M.C.)} = \frac{W_I - W_f}{W_I - W_c} \times 100$$

where

W_I = Initial weight of (crucible + sample)

W_f = Final weight of (crucible + sample) after drying

W_c = Weight of crucible

$$\% \text{ Ash content} = \frac{W_f - W_c}{W_I - W_c} \times 100$$

where

W_I = Initial weight of (crucible + sample)

W_f = Final weight of (crucible + sample) after combustion at 750°C

W_c = Weight of crucible

B. Calorific value

Calorific value of a fuel is determined by calorimetry which depends on the principle that amount of heat lost by a body is equal to the amount of heat gain. This test enables to compare different fuels in their heat producing capacity.

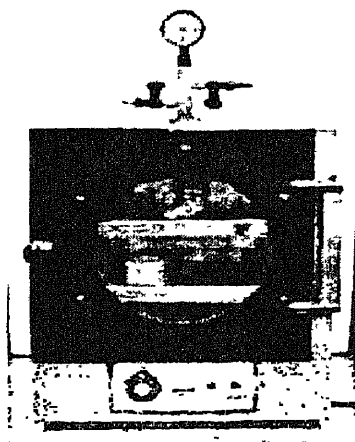


Fig 2: Moisture content analysis
in vacuum oven

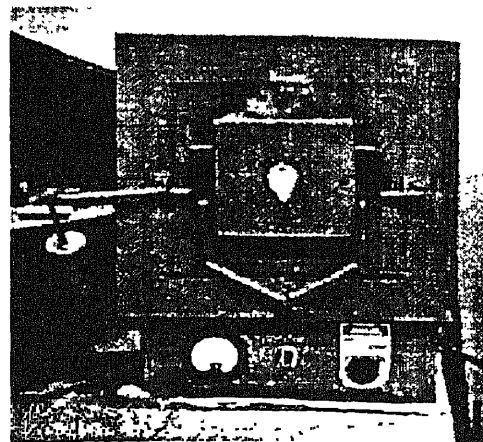


Fig 3 : Ash content analysis in
muffle furnace

Procedure

- 1 Initially the test is done with benzoic acid to standardize the instrument.
- 2 Approximately 1 g of sample is taken and a pellet is made using pellet press.
- 3 Record the weight of crucible.
- 4 Keep the pellet in it and record the combined weight of the crucible and pellet.
- 5 Take 5 cm of cotton thread and record the combined weight of crucible, pellet and thread.
- 6 Take out the thread and put the crucible with pellet in position.
- 7 Cut 6.5 cm of the fuse wire and attach it between the electrodes of the bomb.
- 8 Tie the thread in the centre of the fuse wire and pass it through the crucible in contact with the sample to carry the ignition flame to the sample.
- 9 Place the lid on the bomb and gently tight it. When closing the bomb always make sure that sample is not disturbed and heat gasket on sealing ring are in good condition.
- 10 Connect the high pressure valve in oxygen cylinder outlet.
- 11 Connect copper tube to pressure gauge, pressure gauge to filling tube and filling tube to bomb valve. All connections should be air tight.
- 12 Open the filling connection slowly.
- 13 Observe the gauge continuously while filling oxygen.
- 14 Allow pressure to rise up to 18 atmosphere.
- 15 Close the valve immediately.
- 16 Detach the bomb from oxygen supply.
- 17 Place the bomb in water for check of any leakage.

- 18 Weigh the calorimeter vessel.
- 19 Add 2 kg of distilled water in it.
- 20 Keep the O₂ filled bomb in it.
- 21 Place the whole apparatus in the calorimeter.
- 22 Connect it to the ignition circuit.
- 23 Adjust the stirrer.
- 24 Adjust the Beckman thermometer between 0-1 °C.
- 25 Place the thermometer and cover in position.
- 26 Check the continuity of the circuit by pressing the switch.
- 27 Start the stirrer mechanism.
- 28 Read the temperature after each minute and record the data in preliminary period.
- 29 If the average change of temperature after each minute for five minutes is less than 0.00072 °C, fire the sample.
- 30 Read the temperature after each minute continuously and record the data in chief period.
- 31 Keep recording the data till its rate of change again becomes constant or comes down.
- 32 Remove the bomb from calorimeter after half an hour.
- 33 Release the pressure.
- 34 Measure the length of ignition wire used.
- 35 Fill the data sheet and do calculations as given below.

Data collection

- | | | |
|---|---|-------|
| a) Weight of crucible | = | -- g |
| b) Weight of crucible + thread | = | -- g |
| c) Weight of crucible + thread + pellet | = | -- g |
| d) Length of nicrome wire taken | = | -- cm |
| e) Length of nicrome wire left | = | -- cm |
| f) Room temperature | = | -- |

g)

Preliminary period		Chief period		After period	
Time	Temperature	Time	Temperature	Time	Temperature
1.		t_1			
2.		t_2			
.		.			
.		.			
.		.			
.		.			
.		.			
t_o		t_n			

 t_o - Firing temperature t_n - Temperature after which rate of change of temperature is constant

h) Heat due to fuse wire used =

i) Calorific value of nicrome wire = .335 cal/mg

j) Thread connection (0.005 g) = 21 cal

k) Dickinson correction = $V' (T_a - T_o)$ $V'' (T_n - T_a)$ $V' = -0.0017; V'' = 0.002$ Cooling correction = $V' (T_a - T_o) + V'' (T_n - T_a)$ l) Uncorrected temperature rise = $(t_n - t_o)$

m) Add cooling correction

n) Corrected temperature rise = t_c o) Heat liberated = (Thermal heat capacity) x T_c

p) Subtract thread correction = (Weight of thread x 4.18)

q) Subtract wire correction

For determining apparent heat capacity by Benzoic acid

$$W = \frac{(M \times 6318) + E}{T} = \text{cal}/^\circ\text{C}$$

where

W = Apparent heat capacity

M = Mass of Benzoic acid

E = Correction for heat of combustion of firing wire and thread

$$T = \frac{(\text{Length of fuse wire}) \times 2.45 \times 0.335 + (\text{Weight of thread}) \times 4.18}{\text{Difference in temperature in chief period } (t_n - t_o)}$$

Calculation

$$\text{Calorific value} = \frac{(t_c \times W) - (M + N)}{\text{Weight of sample (g)}} = \text{kcal/kg}$$

where

- t_c = Corrected temperature rise ($^{\circ}\text{C}$)
 W = Apparent heat capacity by Benzoic acid (cal)
 M = Correction for thread (cal)
 N = Correction for nicrome ignition wire (cal)

C. Determination of Carbon monoxide

The CO concentration is measured using gas chromatography, the flame ionization detector along with carbosphere column and methanizer. Nitrogen was used as carrier at a flow rate of 30 ml/min. Oven, methanizer and detector temperatures were 80-150 $^{\circ}\text{C}$, 300 $^{\circ}\text{C}$ and 330 $^{\circ}\text{C}$ respectively. The gas sample was collected in the tedlar bag for analysis. The column gives resolution for CO which is converted to methane by methanizer and detected by flame ionization detector. The area under the peak in the chromatograph relates to the concentration of CO.

Flow rate adjustment

- 1 Connect the soap bubble flow meter to carrier gas outlet pipe with the help of connecting polyethylene tube connected with bubble flow meter.
- 2 Fill the soap solution in the bulb of flow meter.
- 3 Open the gas cylinder and adjust the regulator to give 2 kg/cm² pressure.
- 4 Press the bubble.
- 5 Start the stop-watch as the bubble surface crosses the 0-ml.
- 6 Stop the stop-watch as the bubble surface sweeps the particular volume eg. 15 ml. Note down the time taken for bubble surface to sweep the known volume.
- 7 From above data recorded flow rate is calculated as:

$$\text{Volume flow rate (ml/min)} = \frac{\text{Volume swept by bubble surface (ml)}}{\text{Time taken (min)}}$$

- 1 Adjust the corresponding needle value to get flow rate of 2.5 ml/min.
- 2 Again check the flow rate repeating step 1 to 7.
- 3 Again set flow rate and check it, i.e. repeat step 1 to 8 until it adjusts to required flow rate
- 4 Repeat the same procedure for 2nd column.
- 5 After completing clean the apparatus components for future use.
- 6 To set flow rate of hydrogen, open the cylinder and adjust the regulator to read 2 kg/cm².
- 7 Adjust the needle valve in instrument for hydrogen to give an output pressure of 0.75 kg/cm²
- 8 To set flow rate of air open the cylinder and adjust the regulator to read 2 kg/cm².
- 9 Adjust the needle valve for air to given an output pressure of 0.5 kg/cm².
- 10 Now light the flame.
- 11 Check whether gases has ignited or not.
- 12 Increase air output pressure to 0.75 kg/cm².
- 13 When the work is over, to shut off flame, first close hydrogen supply then air and lastly carrier.

Leakage test

The presence of leak results in the leakage of samples and irregular shift in retention times of sample components. Presence of it is generally established by pressure drop method and exact position of leakage is traced by applying soap solution.

- 1 Block the outlet of exit tube using cap union or merely blocking with finger.
- 2 Raise carrier pressure to about 3 kg/cm².
- 3 After about 2 min. turn off the gas supply by rotating the pressure gauge regulator knob fully anti-clockwise or closing the shut-off vane.
- 4 Pressure gauge reading will decrease a little in beginning but should become steady.
- 5 Observe the pressure gauge. The pressure should not decrease by more than 1 psi in 15 min. (if there is no leakage).
- 6 If leakage is there trace it with soap solution.
- 7 Clean the beaker with water.
- 8 Take water in the beaker to half of its capacity.
- 9 Add a tea spoon full soap in water.
- 10 Stir it with stirrer.

- 11 Turn on the gas supply by rotating the pressure regulator knob clockwise after opening the shut-off vane.
- 12 Apply the soap solution to the suspected points of leakage.
- 13 Observe for bubbles at joints.
- 14 In the event of leakage the relevant nut is tightened by means of a spanner.

Conditioning of column

When a new column or repacked column is installed in column oven it must be conditioned to equilibrate the liquid phase. Bleed off contamination of detector is reduced considerably by conditioning the column. By conditioning low molecular weight impurities bleed out of the column.

- 1 Disconnect the column outlet to the detector.
- 2 Switch on the temperature controller module.
- 3 Set the column oven temperature at 250°C and other temperatures are kept at normal operating temperatures.
- 4 Open the shut off vane and start the carrier supply by rotating pressure regulator knob clockwise till normal operating pressure (flow rate).
- 5 Keep in same conditions for the required time of conditioning.

Septum replacement

A worn septum will result in the loss of sample due to vaporization "blow-back". Every day in morning (after 20-30 injections) septum require replacement.

- 1 Shut off the carrier supply.
- 2 Remove the worn septum.
- 3 Replace with a new one.

Calibration

The column is conditioned before starting the calibration in order to equilibrate the liquid phase. Bleed off contamination of the detector is reduced considerably by conditioning the column. It also bleeds out the low molecular weight impurities. The calibration gas of 262 ppm CO in N₂ was used as standard. This mixture is diluted with nitrogen in the U tubes having silicon septum arrangements. The exact concentration of the prepared mixture was calculated as

$$\frac{\text{volume of standard mixture}}{\text{Total volume gas (standard + nitrogen)}} \times 262$$



Fig. 5 Gas chromatograph used for the CO emission

D. Velocity measurement using Differential Density Manometer

Differential density manometer is used in place of the inclined cum vertical manometer supplied on the panel.

Preparation

The following sequence should be adopted for the filling up of the manometer fluids.

- 1** The manometer is complete in all respect except that the manometer fluids have to be filled into it.
- 2** Before filling the manometer fluids, the system must be levelled. Place the equipment on a reasonably flat surface. Adjust the level by operating the levelling screws provided on the bottom of the panel. Once the correct level is attained, fluids have to be filled into it.
- 3** The syringes have been provided for filling manometer fluids through the two ends of glass tube. The right end of glass portion is connected with +portion through PVC pipe and same way the left end of glass tube is connected with -portion. To filling the fluids both the PVC pipe should be loosen and then take aniline in a syringe and insert the needle of syringe into the + portion at glass tube. In second syringe take distilled water and insert the needle of distilled water filled syringe into the portion of glass tube. Repeat that procedure five, six times, and add both the fluids till its level reaches the zero mark as shown on the scale. If the fluid over shoots the zero mark then note the initial reading. This can be subtracted from actual

reading. For zeroing the manometer reading loosen the drain end and allow the fluids to drip from the drain end until the column is at zero.

The following sequence should be adopted for taking the reading at manometer.

- 1 After filling the manometer fluids connect the two ends of glass tube with PVC Pipe, which are being connected with (+ & -) ends of pitot in wooden panel.
- 2 Now connect the two ends of the manometer marked Pitot (+ & -) to the ends of the pitot tube through Hose Pipe. Ensure that the pitot end, which has a mark, is connected to the +ve end of the manometer.
- 3 Check that the manometer is levelled and note the initial reading if it is not at zero.
- 4 Insert the pitot tube into the stack keeping the marked end the tube facing the air stream.
- 5 Orient the pitot tube axis along the direction of flow often you would have to take the best alignment by gradually rotating the pitot tube in either direction while keeping a watch on the man reading. Best alignment is indicated by the highest reading.
- 6 Note the manometer reading at various transverse points.

In situation where the airflow is turbulent the manometer reading may tend to fluctuate. In such cases the average displacement at each transverse point may be recorded. Excessive oscillation of manometer fluids indicates improper location of sampling port.

It is important to pluck the clearance between the porthole walls and the pitot tube with cotton waste or asbestos wool to prevent excessive airflow through the porthole.

Withdraw and disconnect the pitot tube once the entire cross section of the stack has been transversed.

Calculation

As "S" Type pitot tube is used along with differential density manometer to measure the velocity of air stream inside the chimney or duct. The pitot tube inserted into a stack develop a differential pressure proportional to the kinetic head of the smoke stream and this pressure is measured by the inclined manometer tube. The air velocity can be calculated from the following formula.

$$V = K1 \sqrt{\frac{HxTs}{Tamb}} = m / s$$

$K = \text{pitot constant} = 0.85$

Angle factor $C_1 = 0.4695$

putting known values in above formula

$K_1 = 3.44 \times 0.85 \times 0.685$

$K_1 = 2.0$

where H is in cms

T_s in $^{\circ}\text{K}$

T_{amb} is in $^{\circ}\text{K}$

E. Suspended particulate measurement

Sampling & analysis.

- 1 Sample for particulate concentration and gases shall be done at the same point where velocity measurements were carried out.
- 2 To obtain a representative particulate sample, the sampling should be carried out isokinetically, that is the kinetic energy of the gas stream in the stack should be equal to the kinetic energy of the gas stream through sampling nozzle. The iso kinetic sampling rate should be calculated.
- 3 We have three sampling nozzle (1/8", 1/4" and 3/8"). Calculate the Q_m with all 3 sampling nozzle and select one of them for which the sampling flow rate will be about 20 lpm. If the velocity is less than 1m/s it will be difficult to measure the velocity accurately and keep the sampling flow rate isokinetically. In such cases it will be assumed that the velocity is similar to ambient condition and we can sample with the flowrate about 20 lpm.
- 4 After selecting the suitable nozzle, load the preweighed thimble in the filter holder. For that open the filter holder, loosen the screw, place the preweighed thimble and gently tighten the screw.
- 5 Assemble the sampling train by following the instruction given below.
- 6 Check that the vacuum is connected to the vacuum port of the instrument panel and mains cord is plugged into the mains outlet.
- 7 If we use additional impingers for more than 3 gaseous parameter, use the 6.5 ID, 1 meter long hose to connect the dry gas meter in the series with the gas sampling train. The DGM connectors marked "A" & "B" is appropriately polarised to ensure that the inlet and outlet ports of the gas meter are connected to the correct couplers of the sampling train. If it is not required we can use the impingers provided in the panel box itself and connect the "A" and "B" with PVC or tygon pipe.
- 8 Check the gas sampling impingers are correctly connected. The last impinger

outlet must be connected to the gas sampling connector while the inlet of the first impinger connected to the gas manifold.

CAUTION: A wrong polarity in the impinger connection will cause the absorber solution to be sucked into the flow meters, Dry gas meter and pump assembly and damage the equipment.

- 1 Connect the filter holder assembly with the instrument panel.
- 2 Check that the flow control knobs on both 3 and 30 LPM rotameters are shut off. The valves open by anti clockwise rotation of the knob. Ensure that the valves on the mercury manometer are closed isolating the mercury columns.
- 3 Insert the probe holder into the chimney porthole.
- 4 Start the test after sampling rates have been calculated and train assembled and checked for leakages. When the equipment is ready in all respects, record the initial dry gas meter reading and push the sampling probe carefully into the duct to the point nearest to the back wall. This will allow the probe to cool in hot stack as it comes out, shortening the time required for cooling after the sample is taken. It is advisable to allow the nozzle and filter holder to preheat so that moisture present in the gases does not condense in the filter during initial part of the sampling.
- 5 Open the flow control valve on the 3 LPM flowmeter slowly to set up the gas sampling rate. Open the valve on the 30 LPM flowmeter to set up the calculated flow rate. Always set up flow in the 3 LPM flow meter first and then in the 30 LPM flowmeter. Similarly while terminating the sample, shut off the 30 LPM flowmeter first and the 3 LPM flow meter last.
- 6 Duration of sampling : Deem the run to be of sufficient length if one of the following criteria have been obtained:
 - A minimum of 1 m³ of dry gas has been withdrawn for sampling.
 - The mass of particulate matter amounts to at least 20 percent of the mass of the filtering medium in the sampler.
- 7 Experience and intelligent judgement should be applied in determining the sampling time. Too short a time may give unreliable results and too long a time may cause the resistance of the sampling train to exceed the pumps limits.
- 8 At the completion of test, record the final suction pressure at metering point, close the control valve, turn the direction of probe so that the sampling nozzle is facing down-stream and record the final gas volume and time. Remove the sampler carefully from the flue and plug the nozzle to prevent

the loss of sample.

- 9 Record all the details in the SMK data sheet.

Sample recovery

1. After the sampler has cooled, brush down the dust on the inside of the nozzle carefully into the thimble using a small brush remove the thimble and place it in a dust-tight container for transportation to the weighing room. In case the filter holder is kept outside during the sampling, the dust from the sampling probe before the filter holder should be brushed down into the thimble.
2. Determine the mass of dust collected in the thimble by difference, that is, by weighing the thimble before and after the run. After sampling, cool, dry and again weigh the thimble along with dust maintaining the same conditions as prior to sampling.

Protocol for weighing the Thimbles

Two types of filtration thimbles are available ie. Cellulose filtration thimbles, Glass-microfiber thimbles. Selection of a suitable thimble depends on the flue gas temperature and moisture content. Cellulose filters are hygroscopic. So it has to be used in low moisture condition and up to 150°C. Glass-microfiber filter thimbles can be used in high moisture content and flue gas temperature up to 500°C.

Before use

Weighing the thimbles consists of two main steps.

- (a) Conditioning the thimbles
- (b) Weighing the thimbles

Conditioning the thimbles

- 1 Mark the thimbles neatly.
- 2 Clean and dry a tray or beaker and place the thimbles in it.
- 3 Keep the tray/beaker with thimbles inside the oven and close it properly.
- 4 Switch on the oven, Keep the temperature-adjusting knob to 100°C.
- 5 After 1/2 an hour, switch off the oven and remove the thimbles.
- 6 Clean the desiccator with a clean cloth.
- 7 Place 10-15 thimbles in the desiccator and close it carefully.
- 8 Thimbles are to be desiccated for at least 24 hours.
- 9 After 24 hours the thimbles are ready for weighing.

Thimble weighing

The thimbles have to be weighed in an electronic balance with an accuracy of 10 µg.

- 1 Clean the table and remove all chemicals and possible obstructive materials from near the Mettler weighing balance.
- 2 Clear the exterior and interior portions of the balance using a soft cloth.
- 3 Remove the metal disc and thoroughly clean it with a soft cloth. Place it back.
- 4 Close the two-side windows and top window.
- 5 Level the balance using the two screws at the back. A bubble indicator is located near the metal disc inside the balance.
- 6 Switch on the balance. Allow some time for the display to come on and stabilize.
- 7 The balance has to be operated in the five-digit mode i.e. after the decimal place five zeros should be visible (0.00000).
- 8 Place a small piece of clean wax paper on the metal disc. Wait for a while. The display will indicate the weight of the paper. Press the bar tightly once. After a while the display should show 0.00000.
- 9 With a forceps place the thimble on a wax paper which is on a metal disc. Allow sometime for the weight to be indicated. When the reading becomes constant, the green dot on the extreme left will disappear. Only then the reading should be noted.
- 10 Weigh the thimble twice or thrice to get two consistent readings (i.e. difference of 10 or 20 µg only).

After use

- 1 Desiccate for at least 24 hours and weigh in 5-digit electronic balance.

F. Nitrogen oxides measurement

Applicability

This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO₂) per dry standard cubic meter. Without having to dilute the sample.

Procedures

Sample recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature (T_f) the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5 ml portions of distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1N) drop wise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the PH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its content. Seal the container.

Reagents:

Unless other wise indicated it is intended that all reagents conform to the specifications established by the committee on Analytical reagents of the American Chemical society, when such specification are available. Otherwise, use the best available grade.

For sampling

To prepare the absorbing solution cautiously add 2.8 ml concentrated H_2SO_4 to 1 litre of distilled water mix well and add 5 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation do not expose to extreme heat or direct sunlight.

For sample recovery

- Sodium Hydroxide (1n) Dissolve 40 gm NaOH in distilled water and dilute to 1 litre.
- Water. Distilled to conform to ASTM specification DH93-74. Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

For analysis.

- Fuming sulphuric acid. 15 to 18 percent by weight free sulphur trioxide.
HANDLE WITH CAUTION.
- Phenol white-solid
- Sulphuric Acid concentrate 95%
- *Potassium Nitrate*. Dried at 105 to 110°C (226 to 230°F) for a minimum of 2 hours just prior to preparation of standard solution.
- *Standard KNO₃ Solution*. Dissolve exactly 2.198 g of dried potassium nitrate (KNO₃) in distilled water and dilute to 1 litre with distilled water in a 1,000 ml volumetric flask.
- *Working standard KNO₃ solution*. Dilute 10 ml. of the standard solution to 100 ml with distilled water. One millilitre of the working standard solution is equivalent to 100 micro gram of Nitrogen Dioxides (NO₂).
- Water distilled
- *Phenol disulphonic Acid Solution*. Dissolve 25g of pure white phenol in 250 ml concentrated sulphuric acid on a steam bath. Cool, add 75 ml fuming sulphuric acid and heat at 100°C (212°F) for 2 hours. Store in a dark. Stoppered bottle.

Procedures

Sample recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature (T_f) the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5 ml portions of distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (IN) drop wise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the PH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its content. Seal the container.

Analysis: Note the level of the liquid in container and confirm whether or not any sample was lost during shipment note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or analyze an

aliquate of this, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50 ml volumetric flask, and rinse the container twice with 5 ml portions of distilled water. Add the rinse water to the flask and make up to the mark with distilled water, mix thoroughly. Pipette a 25 ml aliquot into the porcelain-evaporating dish. Return unused portion of the sample to the polyethylene storage. Evaporate the 25 ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenol di sulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml distilled water and four drops of concentrated sulphuric acid. Heat the solution on steam bath for 3 minutes with occasional stirring. Allow the solution to cool. Add 20 ml distilled water, mix well by stirring and add concentrated ammonium hydroxide drop wise with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, then must be removed by filtration (centrifugation is an acceptable alternative) use whatman No. 41 filter paper and filtered is diluted to 100 ml by distilled water. If solids are absent the solution can be transferred directly to the 100 ml volumetric flask and diluted to the mark with distilled water. Mix the contents of the flask thoroughly. And measure the absorbance at the optimum wavelength used for the standards using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of distilled water if the absorbance exceeds.

Spectrophotometer Calibration

Optimum Wavelength Determination: For both fixed and variable wavelength spectrophotometers, calibrating against standard certified wavelengths of 410 nm, every 6 months. Alternatively, for variable wavelength spectrophotometer, scan the spectrum between 400 and 415 nm using a 200 ug NO₂ standard solution. If a peak does not occur, the spectrophotometer is probably malfunctioning and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance for both the standard and samples.

Determination of Spectrophotometer calibration Factor K_c Add 0.0, 1.0, 2.0, 3.0 and 4.0 ml of the KNO₃ working standard solution 100 ug NO₂ to a series of five porcelain evaporating dishes. To each, add 25 ml of absorbing solution. 10 ml distilled water, and Sodium Hydroxide (IN), drop wise, until the pH is between 9 and 12 (about 25 to 35 drops each). Beginning with the evaporation

step, follow the analysis procedure of section 4.1 until the solution has been transferred to the 100 ml volumetric flask and diluted to the mark. Measure the absorbance of each solution at the optimum wavelength.

Annexure-II**Minutes of the meeting between TERI and Guwahati Refinery and action plan**

Project Name: The feasibility of oil sludge based biomass briquettes and their environmental impact assessment as fuel

Members Present**From Guwahati refinery**

1. Prithwiraj Sur, DGM(T)
2. P.K. Goswami, Sr Manager (Energy and safety audit)
3. Amalendu Kumar, Chief Instrumentation Manager

From TERI, New Delhi

1. Sunil Dhingra, Fellow

Date and Venue

11 and 12 January 2001

TERI, New Delhi

Mr. Sunil Dhingra of TERI briefed the team about the Biomass Energy Technology Applications activities and the progress made so far on the project. He explained that there are two types of briquetting technology exist one with binder (in low-pressure machine) and other without binder (in high-pressure machine). He told the members that under the present study, we had successfully made two types of biomass mixed oil sludge briquettes with a screw type briquetting machine having 25% and 33% biomass. The wet briquettes are then sun dried in the open yard and final dried briquettes have calorific value of about 5,300 kcal/kg. The ultimate and proximate analysis of these briquettes is presently being carried out.

Further, he explained the members that these briquettes were tried in conventional cook stoves and found to be not suitable probably due to high air requirement for complete combustion. Meanwhile, these briquettes are

successfully tried out in a downdraft gasifier of capacity 50,000 kcal/h installed at our Gual Pahari campus.

Regarding the emissions, Mr. Goswami informed that study should concentrate on mainly SO_x, NO_x, and PAHs (Poly Aromatic Carbons) and heavy metals like Ni, Vanadium and lead only.

Mr. Goswami informed that analysis of the sludge was carried out by Guwahati Refinery and he will forward the result to TERI.

Mr. Kumar would like to know the analysis of producer gas, flue gases and ash content. In reply Mr. Dhingra informed that this analysis would be carried out in next few weeks.

It has been agreed that TERI will organized a review meeting in the month of February and followed by field visit to gasifier manufacturer.

12 January, 2001

The team visited the TERI Gual Pahari Campus where a demonstration of gasifier on oil sludge briquettes was organized. The team also visited the briquetting plant site where oil sludge briquettes were produced. Mr. Dhingra explained the team about the different process steps, which needs to perform to make the briquettes. Mr. Kumar expressed concern that formation of oil sludge briquettes with saw dust has problem from availability of sawmill waste at Guwahati.

He advised that TERI should try to make briquettes with coal dust, which is plenty available at refinery.

Mr. Dhingra told the members that a follow-up project to prepare the detailed feasibility report of gasification of oil sludge briquettes at Guwahati refinery needs to be considered. The study would look into different aspects like capacity of gasifier system, thermal energy needs at Guwahati refinery, which can be met through gasifier system and followed by implementation

Data sheet for trial experiment in cook stove

General data

Date _____
 Stove used _____
 Fuel type _____
 Instrument used for temperature measurement _____

Experimental location

Initial data of experiment

Initial weight of fuel
Amount of kerosene added
Amount of charcoal added

Weight of pot+lid+thermometer .
Initial weight of water .
Initial temperature of water .

Final data of experiment

Final weight of fuel
weight of ash

Final weight of water

Experimental data

[illegible]

Annexure III

Shriram reports



SHRIRAM INSTITUTE FOR INDUSTRIAL RESEARCH

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TEST CERTIFICATE

NO. 03568

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Visit us at <http://www.shriraminstitute.org>

Issued to

Tata Energy Research Institute,
Darbari Seth Block,
Habitat Place, Lodhi Road,
New Delhi-110003.

J.O No 103-143-0976 ARP
Reg. No 50626
Date 17.04.2001
Your Ref. No.: GC-01 (Rev-02)
Date: AM/PURC/GEN
13.03.2001

Sample Particulars: Kind Attn: Mr. Ruby Wadhwa
Materials

One sample described as "RAW SLUDGE" was received.

TEST RESULTS

S.No	Tests	Results	Protocol
1.	Gross Calorific Value, (Cals/gm)	4340	Bomb Calorimeter
2.	Net Calorific Value, (Cals/gm)	2430	Bomb Calorimeter
3.	Carbon Content, (% by mass)	60	CHN Analyzer
4.	Hydrogen Content, (% by mass)	9	CHN Analyzer
5.	Nitrogen Content, (% by mass)	3.9	Kjeldahl
6.	Sulfur (as S) (% by mass)	0.5	Gravimetric
7.	Ash Content, (% by mass)	3.4	Furnace Ignition
8.	Oxygen Content, (% by mass)	23.2	By Difference

DOR: 13/03/2001

DOC: 16/04/2001

AUTHORISED SIGNATORY

Dy. Director / AD - Chief
Sr. Scientist / Scientist

NOTE 1 The result listed refer only to the tested sample(s) and applicable parameter(s). Endorsement of products is neither inferred nor implied.
2 Total liability of our institute is limited to the invoiced amount.
3 Samples will be destroyed one month from the date of issue of test certificate unless otherwise specified.
4 This report is not to be reproduced wholly or in part and cannot be used as an evidence in the Court of law and should not be used in any advertising media without our special permission in writing.
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Issued to

Tata Energy Research Institute,
Darbari Seth Block,
Habitat Place, Lodhi Road,
New Delhi-110003.

Sample Particulars : Kind Attn: Mr. Ruby Wadhwa
Materials

J.O. No 103-143-0977 ARP
Reg No 50626
Date 17.04.2001
Your Ref. No GC-01 (Rev-02)
AM/PURC/GEN
Date 13.03.2001

One sample described as "Coal Dust And Oil Sludge Briquette" was received.

TEST RESULTS

S.No	Tests	Results	Protocol
1.	Carbon Content, (% by mass)	85.3	CHN Analyzer
2.	Hydrogen Content, (% by mass)	10.6	CHN Analyzer
3.	Nitrogen Content, (% by mass)	1.3	Kjeldahl
4.	Sulfur (as S) (% by mass)	0.5	Gravimetric
5.	Ash Content, (% by mass)	2.3	Furnace Ignition
6.	Oxygen Content,	Nil *****	By Difference

DOR: 13/03/2001

DOC: 16/04/2001

[Signature]
AUTHORISED SIGNATORY

NOTE

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Sr. Scientist / Scientist



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Habitat Place, Lodhi Road,
New Delhi-110003.

Sample Particulars : Kind Attn: Mr. Ruby Wadhwa
Materials

J.O. No 103-143-0978 ARP

Reg No 50626

Date 17.04.2001

Your Ref. No.: GC-01 (Rev-02)

AM/PURC/GEN

Date : 13.03.2001

One sample described as " Biomass And Oil Sludge Briquette" was received.

TEST RESULTS

S.No	Tests	Results	Protocol
1.	Carbon Content, (% by mass)	72.5	CHN Analyzer
2.	Hydrogen Content, (% by mass)	1.4	CHN Analyzer
3.	Nitrogen Content, (% by mass)	0.9	Kjeldahl
4.	Sulfur (as S) (% by mass)	0.4	Gravimetric
5.	Ash Content, (% by mass)	24.8	Furnace Ignition
6.	Oxygen Content,	Nil *****	By Difference

DOR: 13/03/2001

DOC: 16/04/2001

AUTHORISED SIGNATORY

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5 In case any reconfirmation of contents of this test certificate is required please contact our office.

Dy. Director / AD - Chief
Sr. Scientist / Scientist



SHRIRAM INSTITUTE FOR INDUSTRIAL RESEARCH

(A UNIT OF SHRIRAM SCIENTIFIC & INDUSTRIAL RESEARCH FOUNDATION)

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TEST CERTIFICATE

NO. 03571

Issued to :

Tata Energy Research Institute,
Darbari Seth Block,
Habitat Place, Lodhi Road,
New Delhi-110003.

Sample Particulars : Kind Attn: Mr. Ruby Wadhwa
Materials

JO No 103-143-0979

Reg. No 50626

Date 17.04.2001

Your Ref. No.: GC-01 (Rev-02)

AM/PURC/GEN

Date : 13.03.2001

One sample described as "Ash remained After Combustion of Coal Dust And Oil Sludge Briquette" was received.

TEST RESULTS

S.No	Tests	Results	Protocol
1.	Carbon Content, (% by mass)	67.2	CHN Analyzer
2.	Hydrogen Content, (% by mass)	7	CHN Analyzer
3.	Nitrogen Content, (% by mass)	2.5	Kjeldahl
4.	Sulfur (as S) (% by mass)	0.3	Gravimetric
5.	Ash Content, (% by mass)	8.7	Furnace Ignition
6.	Oxygen Content, (% by mass)	14.3	By Difference

DDR: 13/03/2001

DOC: 16/04/2001

[Signature]

AUTHORISED SIGNATORY

NOTE 1 The result listed refer only to the tested sample(s) and applicable parameter(s). Endorsement of products is neither inferred nor implied.
2 Total liability of our institute is limited to the invoiced amount.
3 Samples will be destroyed one month from the date of issue of test certificate unless otherwise specified.
4 This report is not to be reproduced wholly or in part and cannot be used as an evidence in the Court of law and should not be used in any advertising media without our special permission in writing.
5 In case any reconfirmation of contents of this test certificate is required, please contact our office.

Dy. Director / AD - Chief
Sr. Scientist / Scientist

